

TO WHOM IT MAY CONCERN

This is to certify, that there have been collaborative research activities between faculty members of *Government Institute of Forensic Science, Nipatniranjan Nagar, Caves Road, Aurangabad-431004* and *Deogiri College, Aurangabad 431004, MS, India*. The details of the collaborative research activities carried out are as follows:

Collaborators:

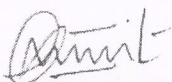
1. Dr. Satish U. Deshmukh, Associate Professor, Department of Chemistry, Deogiri College, Station Road, Aurangabad 431 005, MS, India.

Between

2. Dr. Devidas S. Bhagat, Assistant Professor, Department of Forensic Chemistry, Government Institute of Forensic Science, Nipatniranjan Nagar, Caves Road, Aurangabad-431004, MS, India.

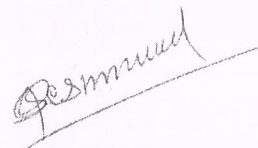
It is hence certified that there have been successful collaboration in term of research and resulted in the research paper publication during 2019-20.

No. of Publications: 01



Dr. Devidas S. Bhagat
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Academic Year 2019-20

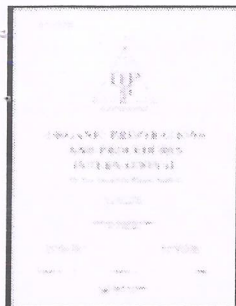
1. Bhagat, D. S., S. U. Tekale, A. K. Dhas, S. U. Deshmukh, R. P. Pawar, and P. S. Kendrekar. "A Rapid and Convenient Synthesis of Acridine Derivatives Using Camphor Sulfonic Acid Catalyst." *Organic Preparations and Procedures International* 51, no. 1 (2019): 96-101.

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A Rapid and Convenient Synthesis of Acridine Derivatives Using Camphor Sulfonic Acid Catalyst

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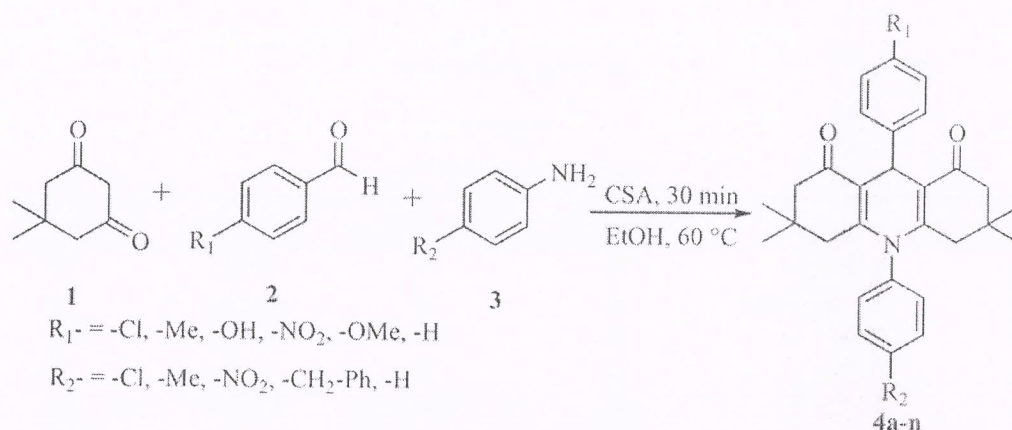
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Scheme 1. Standard model reaction.

Table 1
Effect of Catalyst and Solvents

Entries	Catalysts	Solvent	Time (h/ min)	Temp. (°C)	Yield (%)	Ref.
1	SBA-Pr-SO ₃ H	–	5 min	140	69	19
2	SiO ₂ -I	Ethanol	2–5 h	80	40–90	23
3	TEBAC	Water	10–12 h	100	90	28
4	NH ₂ SO ₃ H	Ethanol	2 h	140	60–75	24
5	BNBTS	–	0.5–2 h	90	80	25
6	CSA	Ethanol	30 min	60	90–94	Present work

Table 2
Effect of Concentration of Catalyst

Entries	Mol %	Solvent	Time (min)	Temp (°C)	Yield (%)
1	No catalyst	Ethanol	30	Reflux	Trace
2	2	Ethanol	30	Reflux	30
3	4	Ethanol	30	Reflux	50
4	6	Ethanol	30	Reflux	60
5	8	Ethanol	30	Reflux	80
6	10	Ethanol	30	Reflux	90
7	12	Ethanol	30	Reflux	90
8	14	Ethanol	30	Reflux	90

10 mol % of catalyst (Table 2, entry 6). In order to evaluate the effect of solvent, reactions were carried out as listed in Table 3. Toluene, acetonitrile, DMF and DCM afforded moderate yields. Methanol and DMSO resulted in good yields; but ethanol furnished the product in 90% yield (Table 3, entry 6), making it the most favorable solvent. To explore the scope of the method, different aldehydes and anilines were treated with dimedone in the presence of 10 mol % of CSA using ethanol as a solvent. All the

apparatus (Optics Technology). The reactions were monitored using thin layer chromatography (TLC) in 40% ethyl acetate:n-hexane on silica gel precoated aluminum foil (Merck). FT-IR (KBr) spectra were recorded at room temperature on a Varian Inova spectrometer, and ^1H NMR spectra in CDCl_3 using tetramethylsilane (TMS) as internal standard on a Bruker Vector spectrometer.

General Procedure for the Synthesis of 3,3,6,6-Tetramethyl-9,10-diphenyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2H,5H)-diones (4)

In a round bottom flask were placed cyclohexanedione **1** (2 mmol), an aromatic aldehyde **2** (1 mmol), an aniline **3** (1 mmol) and CSA (10 mol %) in ethanol (2 mL) and the reaction mixture was stirred at 60°C . The progress of reaction was monitored using TLC. The reaction mixture was quenched with crushed ice and extracted with ethyl acetate (2×15 mL). The organic extract was washed with brine solution (2×15 mL) and dried over anhydrous sodium sulfate, then filtered. The solvent was evaporated under reduced pressure to afford the corresponding crude compounds. The obtained crude compounds were recrystallized from ethanol (Table 4).

Representative Compound Data

9,10-bis(4-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-4-chlorophenylacridine-1,8(2H, 5H, 9H, 10H)-dione (4a): Off white solid; mp. $303\text{--}305^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 0.74 (s, 6H), 0.89 (s, 6H), 1.95–2.14 (m, 8H), 5.15 (s, 1H), 7.08–7.15 (m, 5H), 7.20 (s, 1H), 7.46–7.50 (d, 2H). IR (cm^{-1}) 2950, 1680, 1577, 1491, 650. MS: 492.14 ($\text{M}^+ - 1$).

9-(4-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-10-p-tolylacridine-1,8(2H, 5H, 9H, 10H)-dione (4b): Off white solid; mp. $273\text{--}275^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 0.8. (s, 6H), 0.94 (s, 6H), 1.87 (s, 1H), 2.03–2.17 (m, 7H), 2.49 (s, 3H), 5.23 (s, 1H), 7.06–7.10 (d, 2H), 7.18–7.23 (m, 2H), 7.33–7.39 (m, 4H). IR (cm^{-1}) 2958, 1639, 1574, 1360, 1221, 840; MS: 472.20 ($\text{M}^+ - 1$).

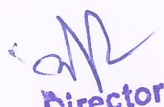
9-(4-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-10-m-tolylacridine-1,8(2H, 5H, 9H, 10H)-dione (4c): Yellow solid; mp. $242\text{--}245^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 0.73 (s, 6H), 0.88 (s, 6H), 1.79 (s, 1H), 1.96–1.99 (m, 2H), 2.05–2.18 (m, 5H), 2.41 (s, 3H) 5.16 (s, 1H), 6.92–6.95 (m, 1H), 7.12–7.19 (m, 3H), 7.25–7.37 (m, 4H); MS: 472.20 ($\text{M}^+ - 1$).

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-(4-nitrophenyl)-10-phenylacridine-1,8(2H, 5H, 9H, 10H)-dione (4k): Faint yellow solid; mp. $278\text{--}280^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 0.79 (s, 6H), 0.96–0.99 (s, 6H), 2.05–2.33 (m, 8H), 5.34 (s, 1H), 7.35–7.39 (d, 2H), 7.50 (d, 1H), 7.58–7.62 (d, 2H), 8.05–8.15 (m, 3H); MS: 469.28 ($\text{M}^+ - 1$).

Acknowledgments

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